Enhanced Low-temperature NH$_3$-SCR Activity over Ce-Ti Oxide Catalysts by Hydrochloric Acid Treatment

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ABSTRACT

The effect of hydrochloric acid treatment on Ce-Ti oxides was investigated for selective catalytic reduction of NO with NH$_3$. The results showed that hydrochloric acid treatment had a positive effect on the low-temperature activity of Ce-Ti oxides. The improved activity of Ce-Ti oxides could be attributed to the increase in the concentration of Ce as well as the amount of Ce^{3+} and chemisorbed oxygen on the catalyst surface. In addition, the enhanced Lewis acidity could improve NH$_3$ absorption, and was also a key factor to enhance the low-temperature activity of Ce-Ti oxides.

Keywords: NH$_3$-SCR; Ce-Ti oxide; Hydrochloric acid treatment; Activity.

INTRODUCTION

Selective catalytic reduction (SCR) of NO$_x$ with NH$_3$ is a mainstream technology for the removal of NO$_x$ emitted from mobile and stationary sources (Xu *et al.*, 2018). V$_2$O$_5$/TiO$_2$ doped with WO$_3$ or MoO$_3$ has been widely used in the past decades (Liu *et al.*, 2019; Wang *et al.*, 2020). Because vanadium is a threat to human health and the environment (Li *et al.*, 2017; Yu *et al.*, 2017), a great deal of attention has been paid to develop an environment-friendly vanadium-free SCR catalyst with high activity.

Cerium-based catalysts are regarded as promising substitutes for commercial vanadium-based catalysts owing to the excellent oxygen storage capacity and redox property of CeO$_2$ (Li *et al.*, 2012a; Jiang *et al.*, 2018). A series of cerium-based SCR catalysts have been synthesized and investigated for the SCR of NO with NH$_3$, such as Ce-Ti oxides (Xu *et al.*, 2008; Gao *et al.*, 2010b; Li *et al.*, 2012b), Ce-W/TiO$_2$ (Chen *et al.*, 2010; Shan *et al.*, 2012; Jiang *et al.*, 2015b), Ce-Cu/TiO$_2$ (Gao *et al.*, 2010a), Ce-Sn-O$_x$ (Li *et al.*, 2013) and Ce-Mo/TiO$_2$ (Liu *et al.*, 2014; Jiang *et al.*, 2015a; Geng *et al.*, 2017). In order to improve the performance of cerium-based catalysts, some researchers tried to treat them with non-metals or add non-metals to them. Gu *et al.* (2010), Zhang *et al.* (2016) and Yang *et al.* (2016b) treated CeO$_2$

with SO$_2$, ammonium sulfate and HF, respectively. All of them found that the treated CeO$_2$ possessed better SCR activity than the untreated one. Yu *et al.* (2016) reported that hydrogen pretreatment could substantially enhance the NH$_3$-SCR activity of Ce-Ti catalyst. Zhang *et al.* (2014) and Jin *et al.* (2017) observed the promotional effect of fluorine additive on the NH$_3$-SCR over CeO$_2$-TiO$_2$ and CeO$_2$(ZrO$_2$)/TiO$_2$, respectively. Concerning the effect of Cl on cerium-based catalysts, there are a few reports. Yang *et al.* (2016a) used NH$_4$Cl as the Cl source and studied the effect of Cl on Ce/TiO$_2$ catalyst. The results indicated that Cl had a negative impact on its catalytic activity due to the inhibition of the adsorption of NH$_3$ and NO$_x$ species. However, in this work, we treated Ce-Ti oxides with different concentrations of hydrochloric acid and found that their activities were improved, especially at low temperatures. Thereafter, a series of characterizations including BET, XRD, XPS and NH$_3$-TPD were performed to provide insight into the substantial changes in Ce-Ti oxide catalysts caused by hydrochloric acid.

EXPERIMENTAL

Catalyst Preparation

The Ce-Ti oxide was prepared by a single step sol-gel method. The mass ratio of CeO$_2$:TiO$_2$ was set as 20:100. A solution of titanium butoxide (TBOT) and anhydrous ethanol was added dropwise to a solution of deionized water, 65–68 wt% nitric acid and cerium nitrate hexahydrate (Ce(NO$_3$)$_3$·6H$_2$O) at room temperature. The molar ratio of the above chemicals was 1:35:19:2:0.1. After vigorously stirred for 3 h at room temperature, the mixture was dried at 80°C for 24 h to form xerogel, followed by calcination at 500°C

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for 5 h in air. The obtained sample was denoted as CT.

The hydrochloric acid treated samples were prepared by incipient wetness impregnation with desired aqueous hydrochloric acid solution on CT. The resulting slurries were left to stand for 24 h and dried at 80°C for 12 h. The HCl-treated catalysts were denoted as CT*x* and *x* represented the molar ratio of Cl and Ce.

Catalyst Characterization

The Brunauer-Emmett-Teller (BET) surface areas of the samples were obtained from N_2 adsorption-desorption isotherms at –196°C using ASAP2020-M (Micromeritics Instrument Corp.). The pore volume and average pore diameter were determined by the Barrett-Joyner-Halenda (BJH) method.

The temperature programmed desorption of NH_3 (NH_3-TPD) was analyzed using a FINESORB-3010 chemisorption analyzer (FINETEC Instruments Corp.) with a thermal conductivity detector (TCD). Firstly, 100 mg of the sample was pretreated at 500°C in He for 1 h and cooled to room temperature. Next, the sample was exposed in a 0.5% NH_3/He (30 mL min^{-1}) gas flow for 1 h and purged with He for 1 h to remove physically adsorbed species. Finally, the sample was heated at a rate of 10°C min^{-1} up to 700°C under He flow.

X-ray diffraction (XRD) patterns were obtained using an X'Pert PRO diffractometer (Panalytical Corp.) with Cu Ka radiation at 40 kV and 40 mA.

X-ray photoelectron spectroscopy (XPS) analysis was conducted on a Thermo ESCALAB 250 spectrometer using monochromated Al Ka X-rays (hm = 1486.6 eV) as a radiation source at 150 W. The binding energy (BE) was calibrated by the C 1s value of 284.6 eV.

SCR Activity Test

The SCR activity was measured in a fixed-bed quartz reactor (i.d. = 8 mm) with 230 mg sample (60–100 mesh). The feed gas contained 1000 ppm NO, 1000 ppm NH_3, 3 vol.% O_2 and N_2 as balance gas. The total flow rate was 500 mL min^{-1}, and the gas hourly space velocity (GHSV) was 150,000 h^{-1}. The concentrations of NO, NO_2 and O_2 were continuously monitored by a 350 Pro gas analyzer (Testo Corp.). The activity data were collected when the SCR reaction had reached a steady state for 30 min at each temperature. NO conversion was calculated by Eq. (1):

$$\text{NO conversion (\%)} = \frac{[NO]_{in} - [NO]_{out}}{[NO]_{in}} \times 100 \quad (1)$$

RESULTS AND DISCUSSION

SCR Activity

Fig. 1 shows the variation of NO conversion as a function of reaction temperature over Ce-Ti oxides treated by different concentrations of hydrochloric acid. It was clear that the treatment by hydrochloric acid could significantly improve the SCR activity of Ce-Ti oxides, especially in the temperature range of 200–350°C. When the molar ratio of Cl/Ce was 5:100, the NO conversion rose from 58.8% to 83.8% at 250°C. In the temperature range of 275–400°C,
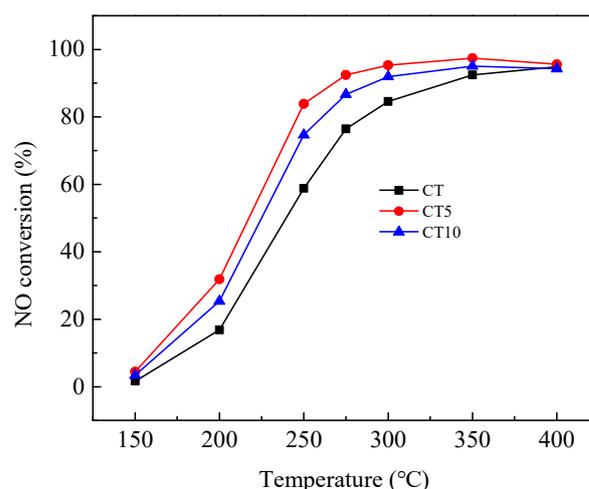

Fig. 1. The SCR activities of different catalyst samples as a function of reaction temperature. Reaction conditions: [NO] = [NH_3] = 1000 ppm, [O_2] = 3%, balance N_2, GHSV = 150,000 h^{-1}.

more than 90% of NO conversion was obtained over CT5. The further increase in hydrochloric acid concentration resulted in the decrease in the catalytic activity. Though the catalytic activity of CT10 was lower than that of CT5, it was higher than that of CT without HCl treatment. These results indicated that hydrochloric acid treatment had a positive effect on the Ce-Ti catalyst for the SCR of NO with NH_3 at low temperature. On the contrary, Yang *et al.* (2016a) found that Cl acted as an inhibitor on the catalytic activity of Ce/TiO$_2$. This might result from the difference in the interaction between different Cl sources and Ce/TiO$_2$.

BET and XRD Analysis

The physical properties of the various samples are listed in Table 1. The BET surface area decreased after hydrochloric acid treatment. The pore size distributions of three samples were measured from N_2 desorption isotherm using cylindrical pore model (BJH method) and shown in Fig. 2. CT yielded the pore diameter distribution centered at about 5.16 nm, while CT5 and CT10 illustrated smaller pore distributions with average pore diameters of 5.08 nm and 5.03 nm, respectively. It could be also seen that the pore volume of the small pores reduced and that of the large pores increased slightly after hydrochloric acid treatment. However, the total pore volume decreased. It meant the block of the small pores or the agglomeration of the catalyst during the treatment process of HCl. Wang *et al.* (2015) found the similar phenomena in the study on the effect of F or Cl on Mn/TiO$_2$ catalyst. Although BET surface area and total pore volume decreased, the SCR activity increased after the treatment of hydrochloric acid. It suggested that the BET surface area was not a main factor for the enhanced SCR activity of CT after hydrochloric acid treatment.

The XRD patterns of different samples are presented in Fig. 3. Only the diffraction peaks ascribed to anatase TiO$_2$ were detected, while the characteristic peaks of CeO$_2$ were not observed. This demonstrated that the treatment by

Table 1. Physical properties of the three catalyst samples.

samples	BET surface area ($m^2 g^{-1}$)	Total pore volume ($cm^3 g^{-1}$)	Average pore diameter (nm)
CT	112.42	0.170	5.16
CT5	103.31	0.140	5.08
CT10	94.81	0.144	5.03
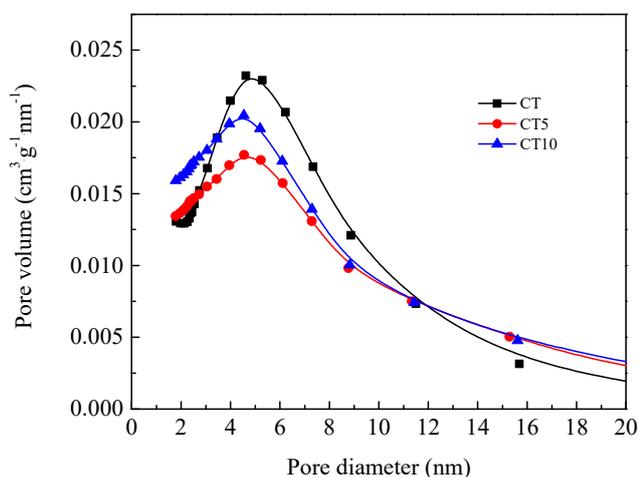
Fig. 2. Pore size distributions of catalyst samples.
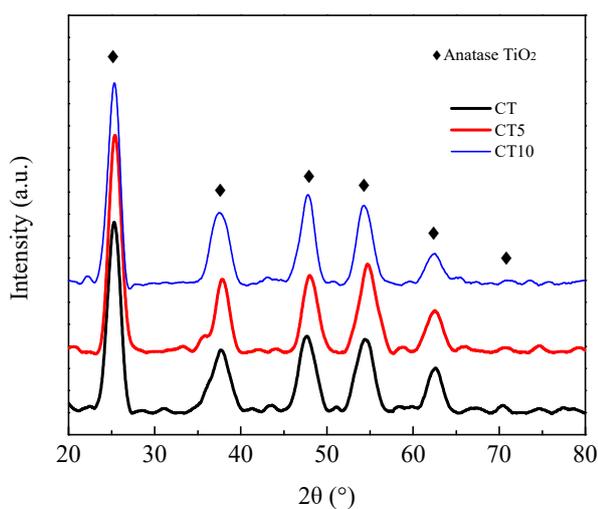
Fig. 3. XRD patterns of the catalyst samples.

hydrochloric acid had no impact on the dispersion of CeO_2 on the surface of TiO_2. CeO_2 was still highly dispersed and existed as amorphous or highly dispersed species in CT. However, the intensity of the diffraction peaks due to anatase TiO_2, especially the peak at $2\theta = 25.3°$, was found to increase slightly after CT was treated by hydrochloric acid. It might be due to the fact that the intercalation of Cl^- into TiO_2

lattice might cause the expansion of TiO_2 lattice since the radius of Cl^- is much larger than that of Ti^{4+} (Wang *et al.*, 2015).

XPS Analysis

Table 2 presents the concentration of various atoms on different samples. The hydrochloric acid treatment led to the increase in the concentration of reactive Ce atoms on the catalyst surface from 2.1% to 3.2%, which was in line with the catalytic activity. However, the concentration of Cl on the catalyst surface (5.2%) was found to be far lower than its nominal value. This might be due to its extraordinarily high volatility (Wu *et al.*, 2013).

Fig. 4 shows the Ti 2p XPS spectra of CT and CT5. The binding energies (BE) of Ti 2p1/2 and Ti 2p3/2 were about 464.2 and 458.5 eV on CT. It suggested that Ti existed in its highest oxidation state (IV) (Fang *et al.*, 2007). After CT was treated by hydrochloric acid, its BE values of Ti 2p shifted to higher ones. It is known that the Ti 2p BE values of Ti^{3+} are 1.8 eV lower than those of Ti^{4+} (Fang *et al.*, 2007). Therefore, Ti still existed as Ti^{4+} on CT5. It should be noted that the intensity of Ti 2p peaks decreased obviously after the treatment by hydrochloric acid. This might be attributed to the fact that an interaction between hydrochloric acid and TiO_2 led to the intercalation of Cl^- into TiO_2 lattice, which was in line with XRD results.

The deconvoluted Ce 3d XPS results of catalyst samples are shown in Fig. 5. The peaks labeled u, u'', u''' and v, v'', v''' are assigned to Ce^{4+}, while u' and v' are attributed to Ce^{3+} (Chang *et al.*, 2013a; Devaiah *et al.*, 2014). Compare with CT, the intensity of the Ce^{3+} peaks increased remarkably over CT5. The ratios of Ce^{3+}/Ce, calculated by the area of the corresponding characteristic peaks, were 28.9% and 37.0% over CT and CT5, respectively. It indicated that the treatment by hydrochloric acid could increase the amount of Ce^{3+} and decrease the amount of Ce^{4+} on the catalyst surface. This might be because the interaction of hydrochloric acid and Ce species promoted the transformation of Ce^{4+} into Ce^{3+}. It is widely accepted that Ce^{3+} could create a charge imbalance and form oxygen vacancies and unsaturated chemical bonds, which is helpful for the formation of chemisorbed oxygen on the catalyst surface (Guo *et al.*, 2015).

After a curve-fitting procedure in Fig. 6, the XPS spectra of O 1s could be deconvoluted into two peaks for CT. The peak at 529.8 eV could be assigned to lattice oxygen (denoted

Table 2. Surface element Concentration of different elements on the samples.

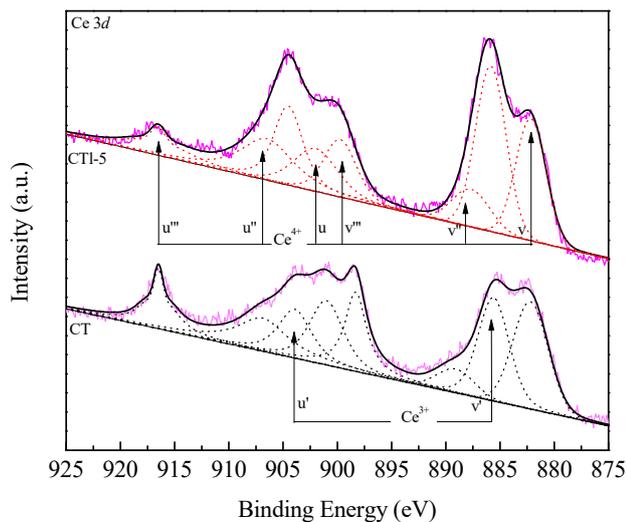| samples	Surface Atomic Concentration (%)				O_β/O	Ce^{3+}/Ce
	O	Ti	Ce	Cl		
CT	65.7	32.2	2.1	/	28.1	28.9
CT5	63.0	28.6	3.2	5.2	32.6	37.0

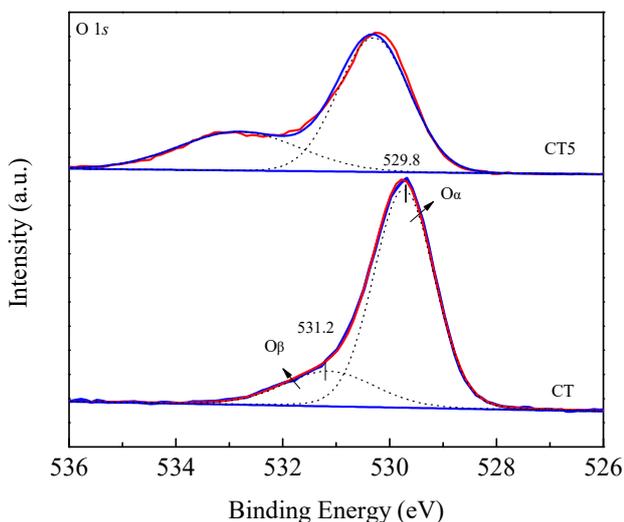**Fig. 5.** XPS spectra of Ce 3d for the catalyst samples.

Fig. 6. XPS spectra of O 1s for the catalyst samples.

as O_α), the other one at 531.2 eV was related to chemisorbed oxygen (denoted as O_β) (Liu *et al.*, 2009; Peng *et al.*, 2013). However, as for CT5, the BE values of O_α and O_β moved to higher ones. The ratio of O_β/O, calculated by the area of the corresponding peaks, increased from 28.1% to 32.6% after the treatment of hydrochloric acid. O_β was considered to be more active than O_α due to its higher mobility, which contributed to nitric oxide adsorption and particularly the oxidation of NO to NO_2 (Chang *et al.*, 2013b). This could be beneficial to improve the SCR activity at low temperature (Zhang *et al.*, 2014).

NH₃-TPD Analysis

NH₃-TPD analysis was performed to study the surface acidity of the catalyst samples and the results were presented in Fig. 7. Each catalyst sample contained a broad NH₃ desorption peak, which could be assigned to the weakly absorbed NH₃ on the Brønsted acid sites (Sang *et al.*, 2012; Zhao *et al.*, 2016). The other small peak centered at about
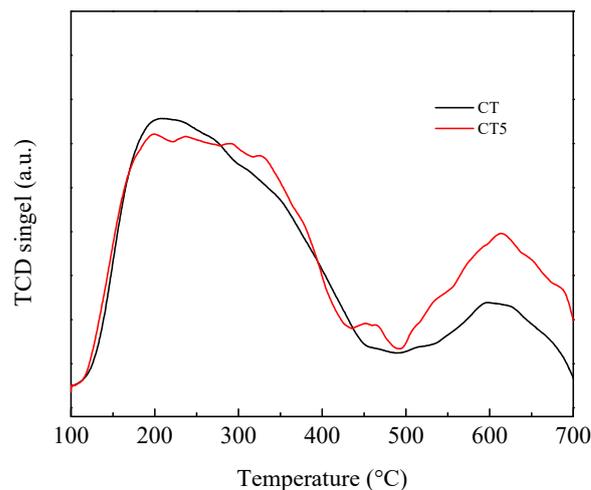

Fig. 7. NH₃-TPD profiles of the catalyst samples.

600°C was associated with the strongly absorbed NH₃ on the Lewis acid sites (Sang *et al.*, 2012; Zhao *et al.*, 2016). After HCl treatment, there was almost no change on NH₃ desorbed on the Brønsted acid sites, while a noticeable increase in NH₃ desorbed on the Lewis acid sites. It is accepted that the adsorption of NH₃ on catalyst surface is greatly dependent on the surface acidity and is crucial for the SCR reaction. Therefore, the enhancement of Lewis acidity should be responsible for the improved SCR activity of CT.

CONCLUSIONS

In this work, the influence of hydrochloric acid on the selective catalytic reduction of NO with NH₃ over Ce-Ti oxide catalysts was investigated. The experimental results showed that the treatment of hydrochloric acid had a promoting effect on the SCR performance of Ce-Ti oxide catalyst. The hydrochloric acid treatment could lead to the increase in the concentration of Ce as well as the amount of Ce^{3+} and chemisorbed oxygen on the catalyst surface, which was beneficial for the enhancement of the SCR activity at low temperature. Furthermore, the enhancement of Lewis acidity should be also responsible for the improved SCR activity of CT.

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